



NASA Aeronautics Research Institute

Scintillating Quantum Dots for Imaging X-rays (SQDIX) for Aircraft Inspection

NASA Aeronautics Research Mission Directorate (ARMD)

2014 Seedling Technical Seminar

February 19–27, 2014



SQDIX - For Aircraft Inspection Acknowledgements

NASA Aeronautics Research Institute

Work was performed under ARMD 2013 Phase 1
at NASA LaRC Non-Destructive Sciences Branch

Primary Investigator: Mr. Eric Burke (NASA LaRC)

Co-PI: Dr. Phillip Williams (NASA LaRC)

Mr. Stan Dehaven (NASA LaRC)

Dr. Mool Gupta (NIA, UVA)

Mr. Dennis Waldron III (NIA, UVA Phd Candidate)

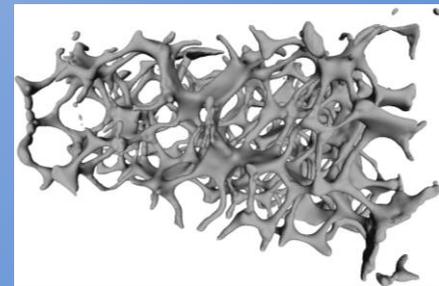
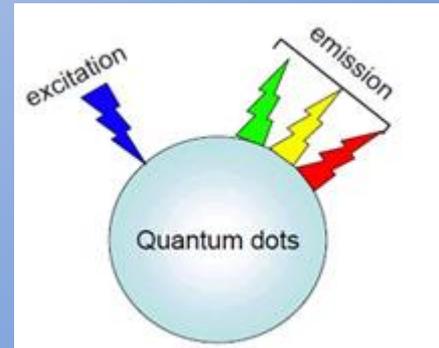
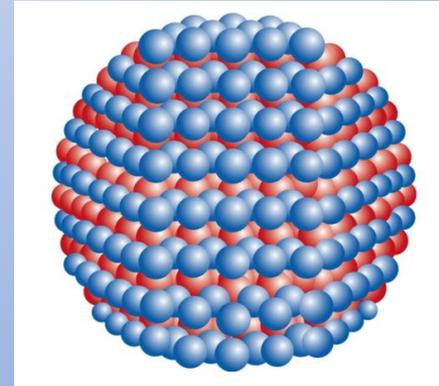
Mesolight LTD.



Overview

NASA Aeronautics Research Institute

- Scintillating Quantum Dots (StQDs) are nano-scale particles that convert X-Ray energy into visible light.
- Recent small scale commercial availability opens the door to developments in X-Ray imaging.
- The use of solution, polymer and thin film processing techniques will enable production of low cost high resolution X-Ray imaging devices.
- X-Ray imaging is a critical inspection method for detecting flaws in aerospace materials and structures.
 - X-Ray imaging is commonly used to detect cracks, voids, delamination, contamination, quality assurance, and perform Computed Tomography (CT).
- X-Ray imaging is also commonly used in the medical community to detect cancer, broken bones, foreign objects and CT.





Overview

NASA Aeronautics Research Institute

- X-Ray detection is also a common element in security applications.
 - X-ray detectors are used to screen for objects such as explosives or weapons. Thousands of pieces of luggage are scanned at airports every day.
 - Large scale drive by scanners have been implemented at ports to scan containers.
- StQDs also exhibit very high X-Ray absorption characteristics. Use of QDs as radiation shielding could lead to easy to apply self-illuminating radiation shielding.
- Computed tomography is also widely used for QA and reverse engineering.





Advantages of QDs

NASA Aeronautics Research Institute

- Fast fluorescent decay time: high frame rate
- High stopping power means greater sensitivity and lower dose required for detection: good for medical applications
- Many scintillators limited by crystal size
- Better room temperature energy resolution
- Higher quantum efficiency
- Inexpensive matrix materials
- X-, α -, γ -ray, and neutron detection demonstrated



Objectives of Phase 1

NASA Aeronautics Research Institute

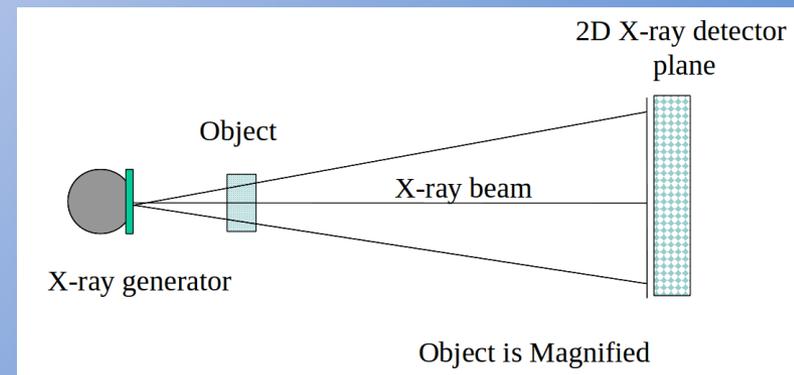
1. Work with commercial entity to acquire large enough quantity of StQDs for experimental characterization and optimization of their material properties for X-Ray imaging.
2. Use X-Ray characterization equipment located at NASA LaRC Non-Destructive Sciences Branch (NESB) for characterization of the StQDs for use as an imaging scintillator.
3. Explore and characterize StQDs in liquid, embedded fiber optic and polymerized forms.
4. Demonstrate the feasibility of the use of StQDs to produce x-ray images (SQDIX).
5. Final report and presentation.



Background

NASA Aeronautics Research Institute

- X-Rays pass through the object being imaged and strike a scintillator layer which creates an image.
- Due to the impracticability of focusing x-rays, the sensors have exactly the same size as the image they capture.
- Typical dimensions of detector pixels are 0.2 mm. In addition there is an Linear diode array (LDA) configuration.
- Magnification is achieved through geometric changes to the set up.
- Many current setups are dependent on growth of exotic materials that are expensive and difficult to handle. (sodium iodide, gadolinium oxysulfide or caesium iodide)





Impact of the Innovation

NASA Aeronautics Research Institute

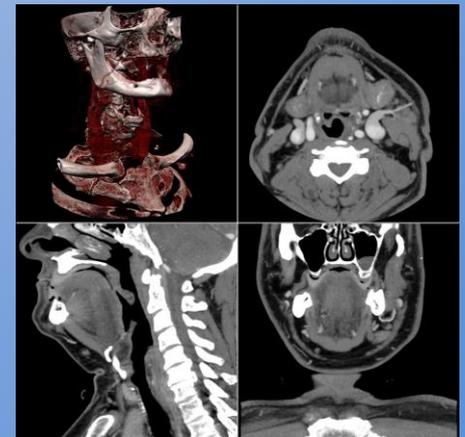
- Development SQDIX has the potential to greatly improve NASA's ability to use x-ray as a inspection method.
 - This directly addresses the Aviation Safety challenge in the 2010 National Aeronautics R&D Plan to monitor and assess the health of aircraft more efficiently and effectively.
 - Development of SQDIX will lower the cost to produce high quality high resolution x-ray panels for field deployment.
 - SQDIX will enable better inspection, allowing for variable magnification without repositioning.
 - Higher sensitivity should enable better characterization of microcracking in both metals and composites.
 - Resolution and sensitivity improvements will directly affect NASA's ability to perform inspection using 3D computed tomography.
 - High sensitivity and the fast refresh rate will enable NASA to take x-ray images of parts in service.
 - Use of quantum dots allows for the use of existing visible light optics and cameras for x-ray inspection, thus greatly reducing production cost.
- Due to the high x-ray attenuation coefficients the use of quantum dots as easily deployable x-ray shielding is possible. Shielding of this type would be visibly illuminate in the presence of radiation.



Impact of the Innovation

NASA Aeronautics Research Institute

- Development of the SQDIX system will greatly impact the medical community
 - In medical x-ray imaging, the amount of exposure to the patient is kept to a minimum due to the ionizing affects of x-ray radiation. Due to the increased sensitivity of the SQDIX system, the time required for exposure to ionizing radiation will be reduced.
 - Higher spatial resolution will enable better characterization of anomalies.
 - The ability to vary the resolution of the x-ray image with out the need to reposition the patient could open new avenues to characterization, including real-time variable focusing x-ray during surgery.
 - Resolution and sensitivity improvements will directly affect the medical community's ability to perform scans using 3D tomography.

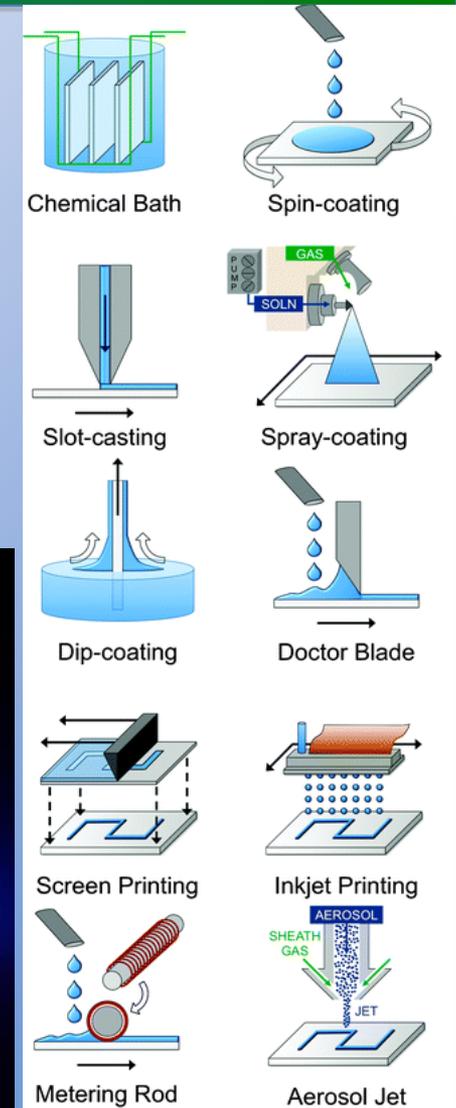




Impact of the Innovation

NASA Aeronautics Research Institute

- Starting with StQDs in solution (toluene) use of polymer and chemical thin film deposition techniques can now be applied.
- There are a variety of techniques that can be applied including spin coating and metered rod deposition.
- This list also includes several techniques that are commonly used in 3D printing which could allow for high volume production.
- Current state of the art x-ray panel production is very dependent on difficult processing methods. Use of solution processing with StQDs will greatly reduce this cost while increasing the quality of the final detector.

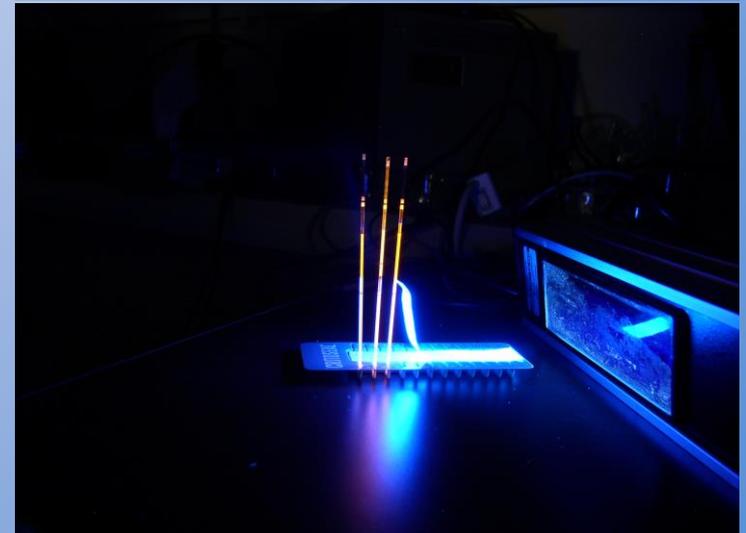
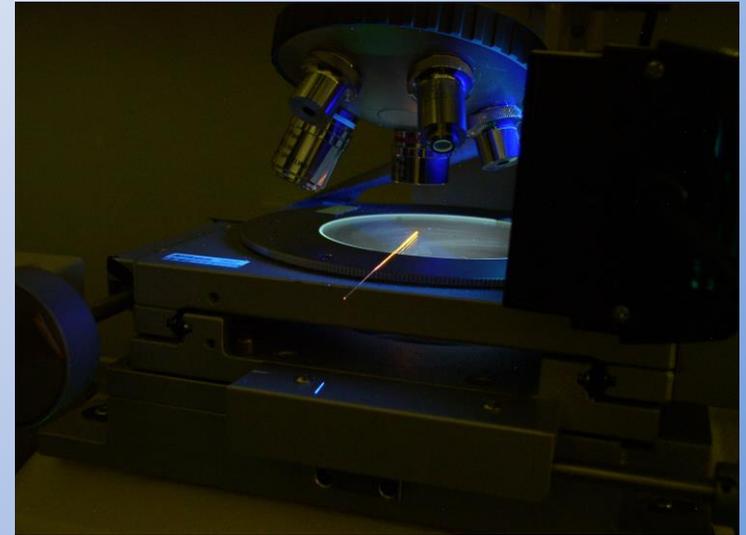




Testing Setup – UV

NASA Aeronautics Research Institute

- Types of QD's
 - Manganese Doped Zinc Sulfide ZnSMn QD's
 - Typical Diameter of ZnS dots are 13–20 nm.
 - Lead Selenide PbSe QD's
- Lamp wavelengths are 365 nm and 254 nm.

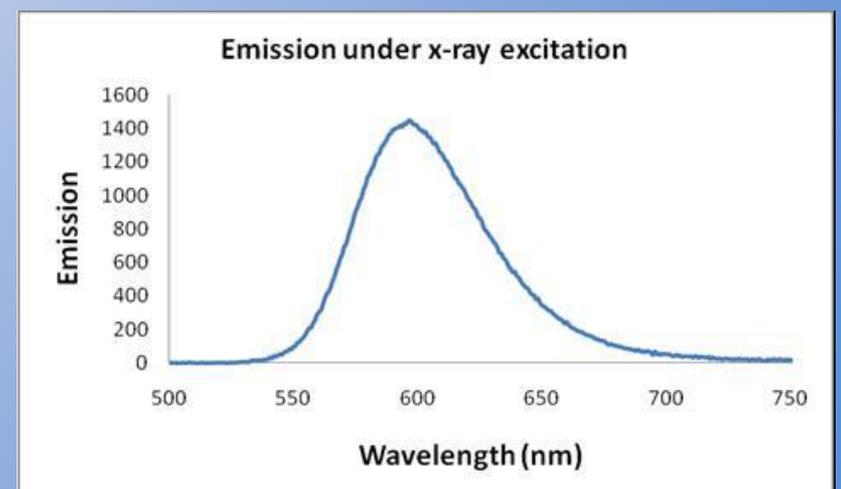
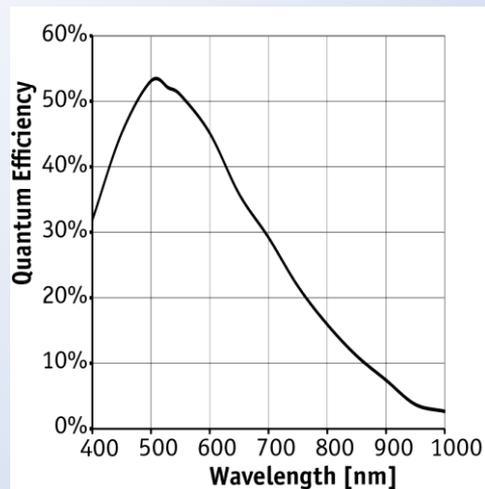
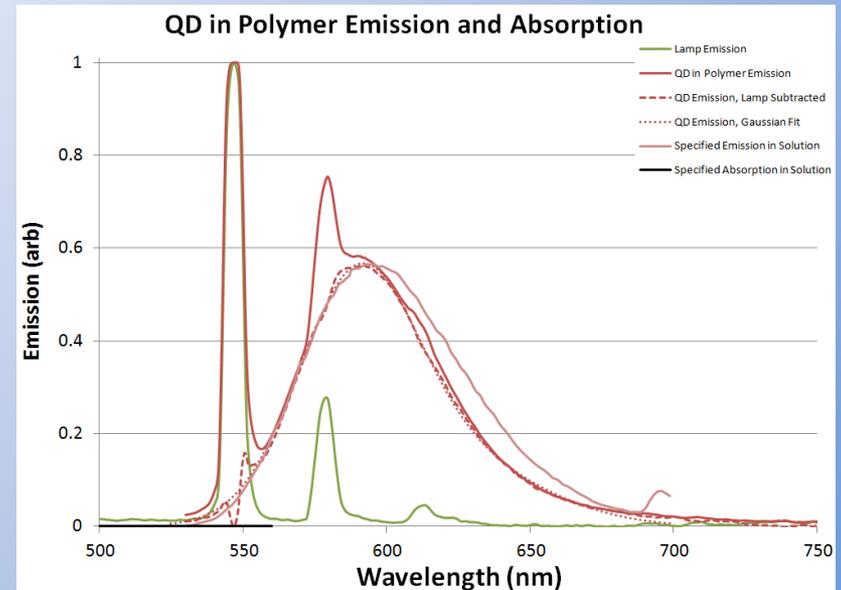




Manganese Doped Zinc Sulfide QD's

NASA Aeronautics Research Institute

- ZnSMn QD's have a peaked orange emission at 585 nm.
- This was verified with both UV and X-Ray Excitation and can be seen in the charts to the right.
- Camera selection was based on the peak emission spectrum data.
- Key Camera Specs – 14 bit digitizer, Ethernet, POE and variable exposure.

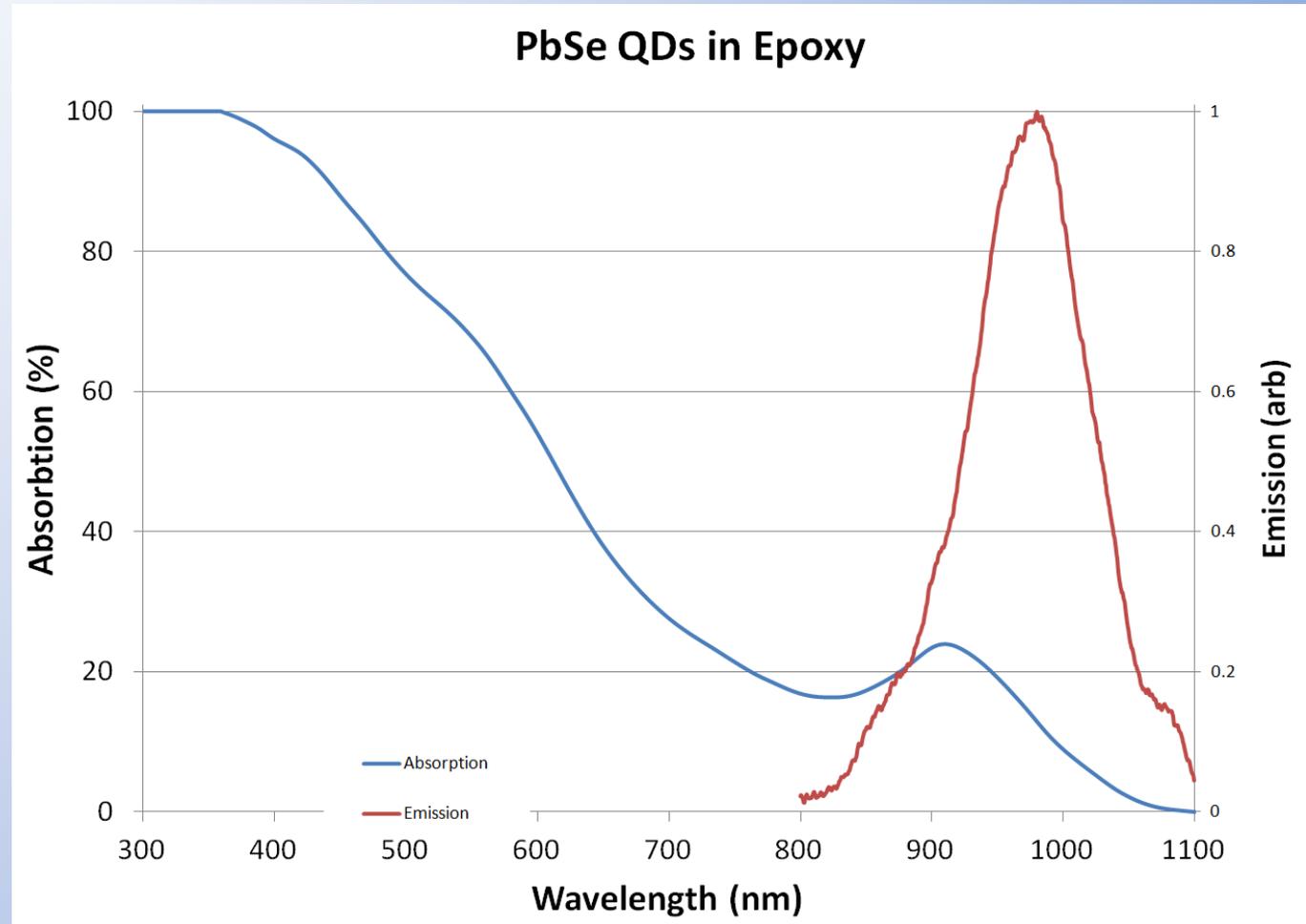




PbSe Qd's

NASA Aeronautics Research Institute

- PbSe QD's emit in the infrared range. But range of excitation frequencies is broader.
- Could be useful for x-ray energy and material composition characterization .

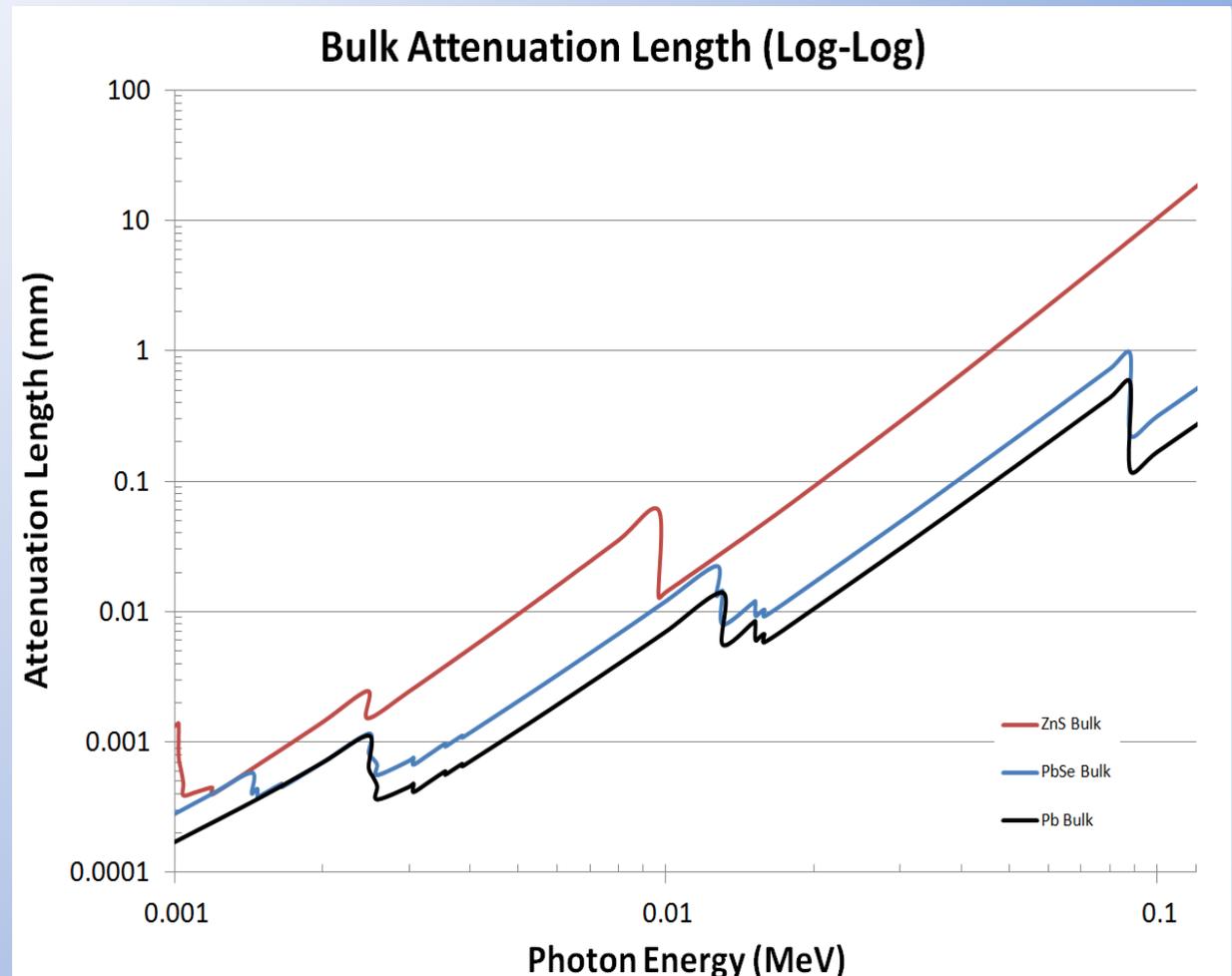




Calculated Bulk Attenuation

NASA Aeronautics Research Institute

- QD's can also have applications in radiation shielding.
- Attenuation length is the amount of material that would be required to reduce the radiation at a given energy by 63.21%.
- In simulation both ZnS and PbSe perform well as x-ray attenuators.
- Also if used as radiation attenuating paint it could visibility glow under excitation.





Solution StQD's Under UV

NASA Aeronautics Research Institute

- Several concentrations of ZnSMn QDs were obtained from Mesolight.
- Concentrations of 10%, 30% and 50% in toluene are shown in the figure here.
- All concentrations visibly fluoresce under UV Excitation.
- The use of UV excitation reduces ionization concerns for radiation workers.”

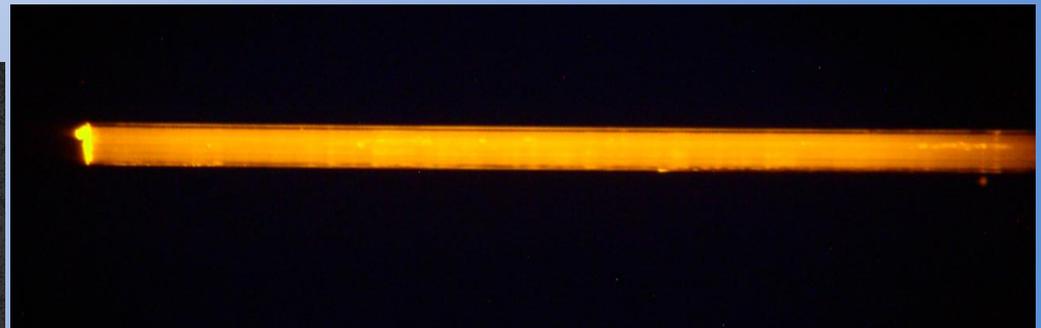
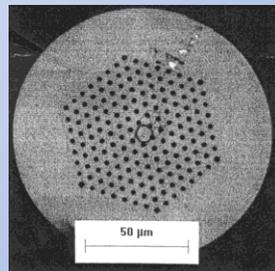
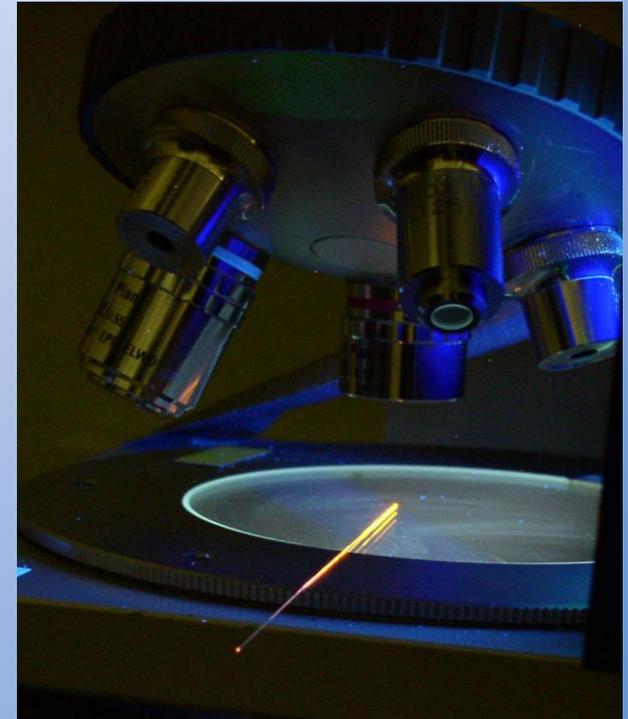
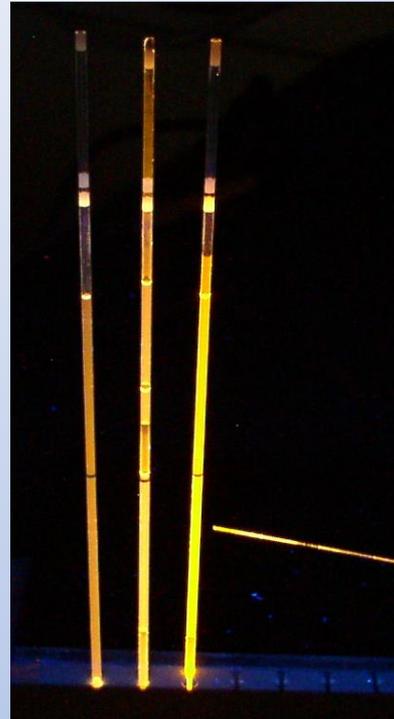




Fiber Optics Under UV

NASA Aeronautics Research Institute

- Several concentrations of ZnSMn QDs were pulled into several diameters of tubing.
- Quantum dots in micro structured (MS) fiber are also being demonstrated here. QDs were easily pumped into the fiber under small pressure gradient.
- A fiber of this type could be mated to a stack light fiber to carry emission to a detector.
- MS fiber outer diameter is 125 μ m and has 168 channels 2.5 μ m Dia.

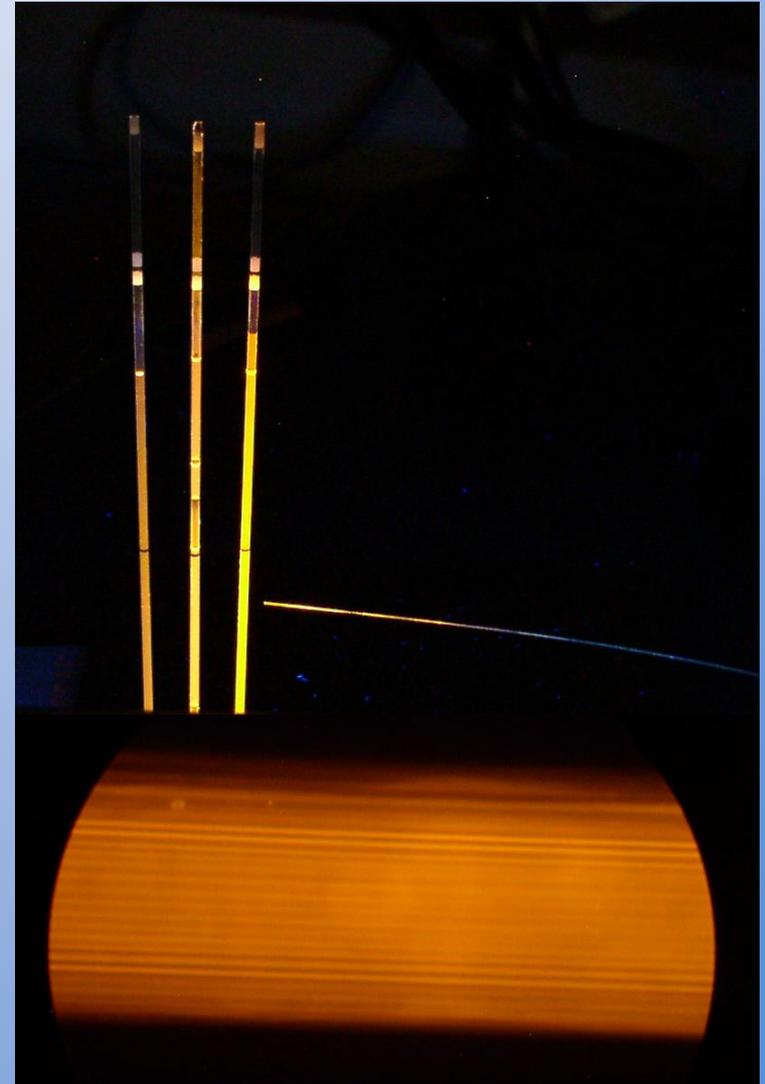




Fiber Optics Under UV

NASA Aeronautics Research Institute

- QD solution was easily placed in a MS fiber though small pressure differential.
- This configuration is highly advantageous for formation of a linear array detector or culminated detection.
- Long fibers can be created through direct coupling or filling the remaining fiber with optical couplet and fusing it to a solid fiber.
- Image of the fiber in the lower right is taken at 500x

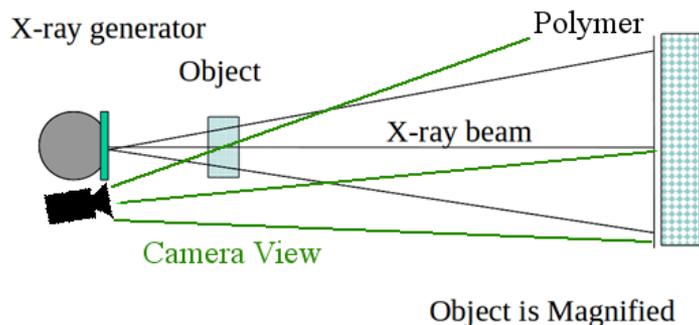
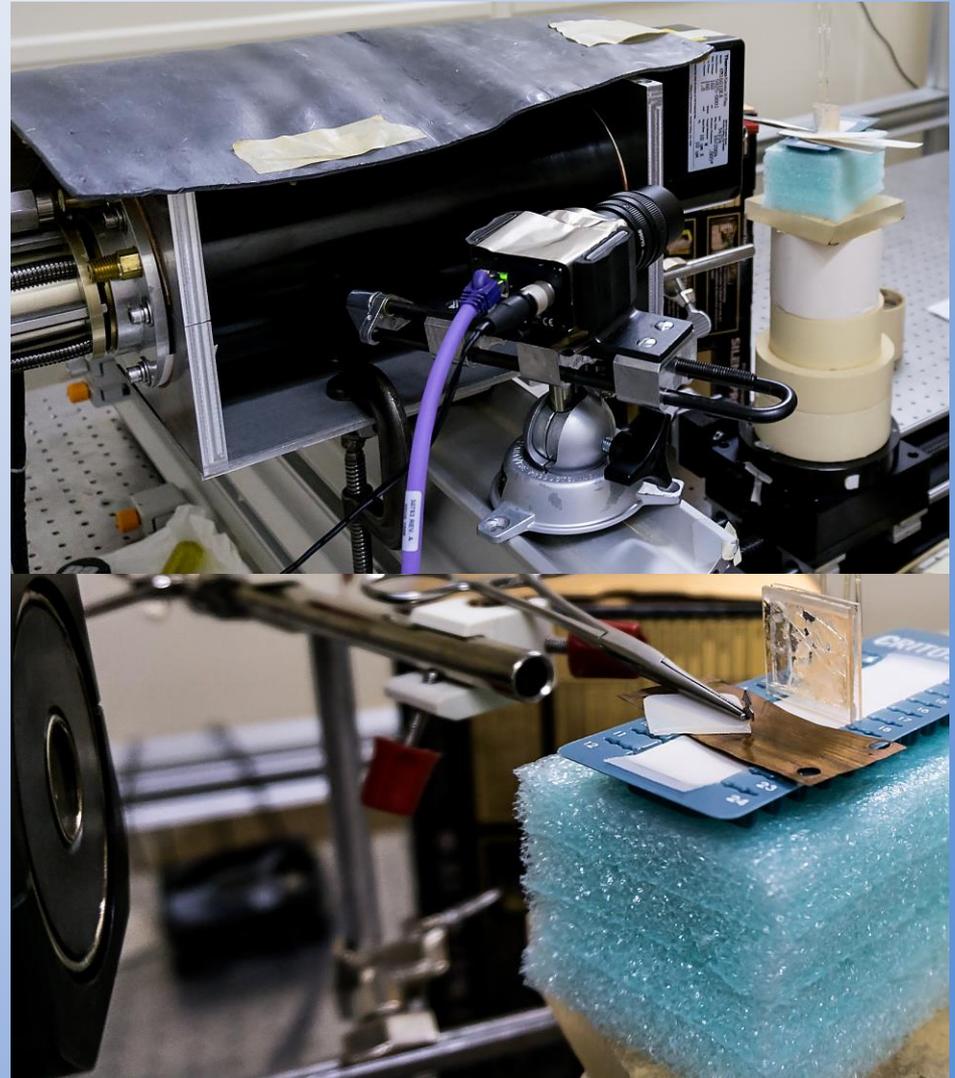




Testing Setup – X-Ray

NASA Aeronautics Research Institute

- Source located at NESB is a micro focus x-ray generator capable of 150 Kv excitation.
 - X-Ray response images were taken at 30, 60, 90, 120 and 150 Kv energy level to quantify response.
- Camera view is approximately in line with the x-ray beam. In some images both the object and the QDs will be visible.

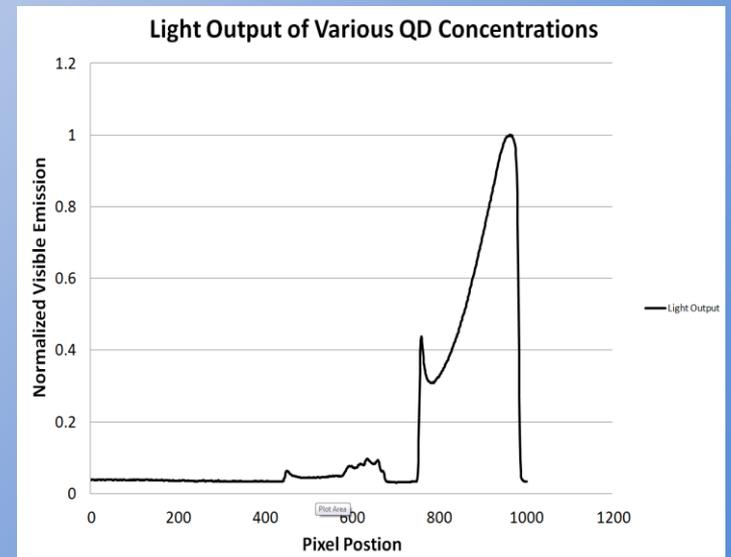
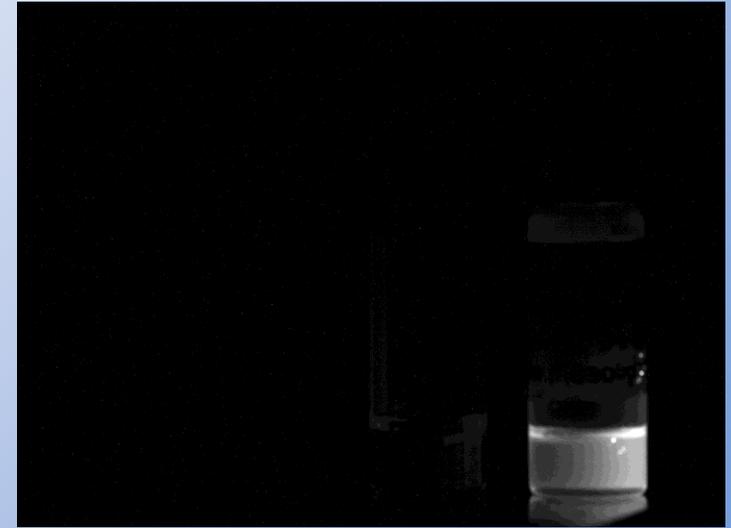




Solution StQD's Under X-Ray

NASA Aeronautics Research Institute

- Initial tests clearly show StQD fluorescence is effected by concentration.
- Under 90 Kv excitation 10% concentration showed no detectable photon count.
- But 30 and 50% concentrations provide detectable emissions under x-ray.
- Response under x-ray excitation appears to be non-linear.
- 50% Concentrations appear to produce 85% more detectable photons than 30% concentrations.
- This observation is critical: Some forms of QDs exhibit a self-annihilation feature of emission and re-absorption at high concentrations. This is clearly not the case for ZnSMn QDs.
- Understanding of the nonlinear response with relation to concentration is still not clear.

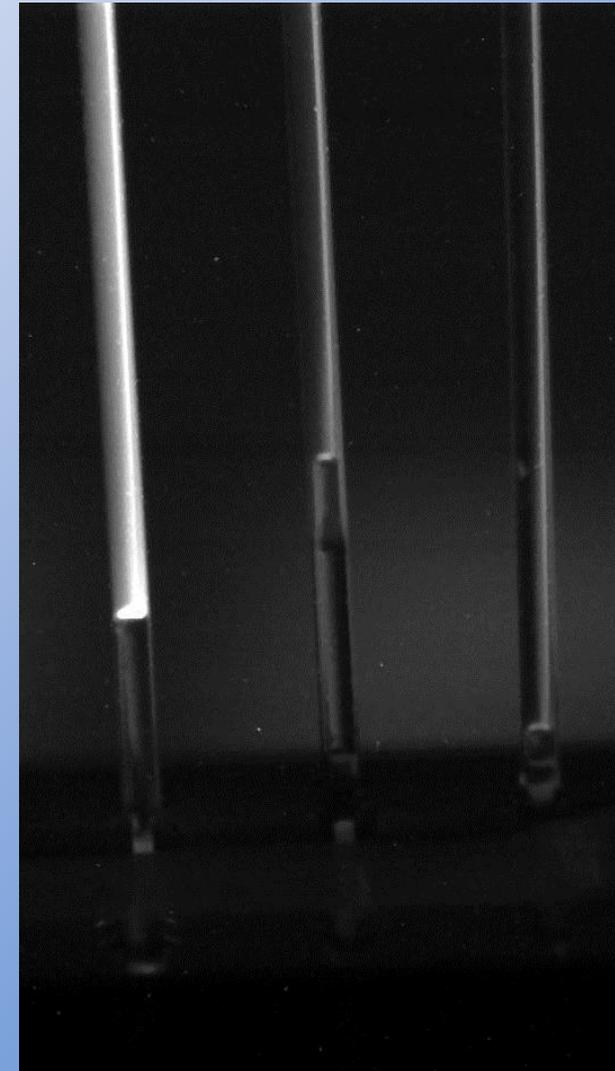
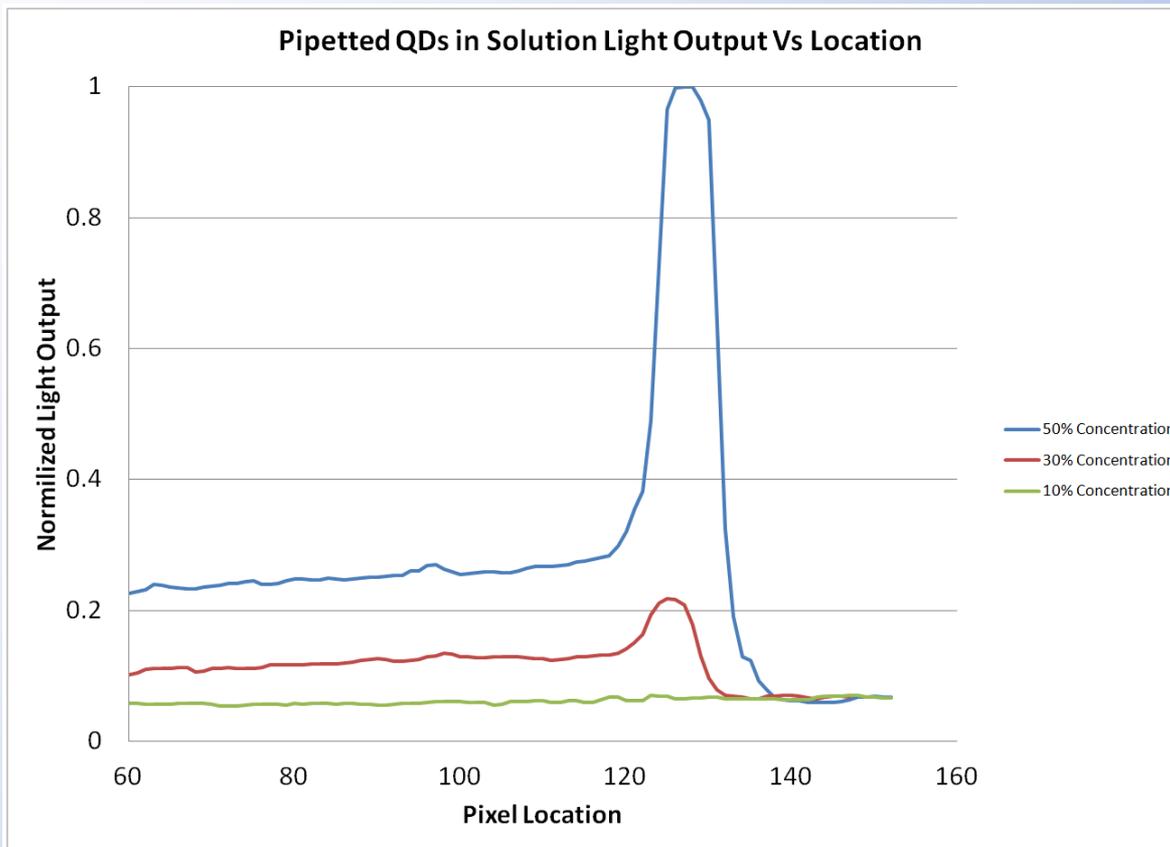




Pipette QD Under X-Ray

NASA Aeronautics Research Institute

- Light output vs concentration effect clearly visible in pipetted QD solution.
- But in addition crystallized QDs shows another jump in light output.

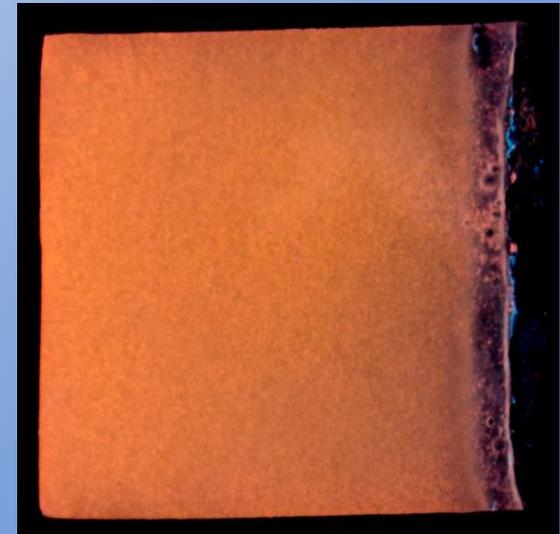




Results – Polymers UV

NASA Aeronautics Research Institute

- Several attempts to create a quantum dot impregnated polymer were attempted.
- Two techniques were used:
 - One method used an outer polymer form to grow QD crystals in the form cavity. Initial attempts at this form creates the broken crystal matrix seen on the far right.
 - Second method is to impregnate polymer prior to cure. Polymer is then cured using UV. This creates a cloudy sample that needs further characterization.

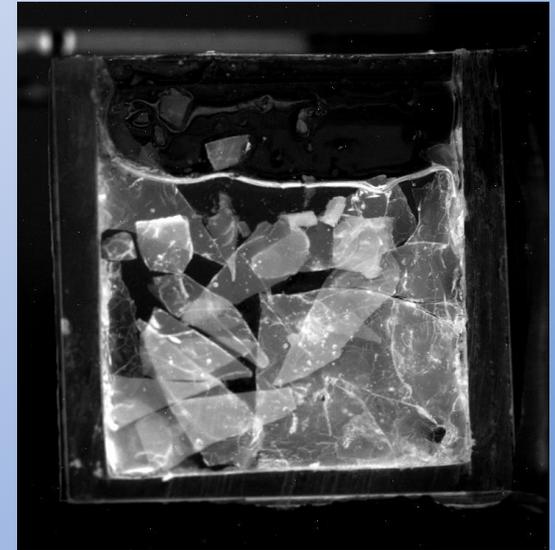




Results - Polymers

NASA Aeronautics Research Institute

- Polymer form sample produced good scintillation under x-ray excitation.
- Non-uniformity in this sample makes imaging with this sample difficult.
- Lower right hand corner of the sample is uniform enough to attempt an imaging application.
- An attempt to deal with the non-uniformity via image subtraction was also attempted and shown on the following slide.



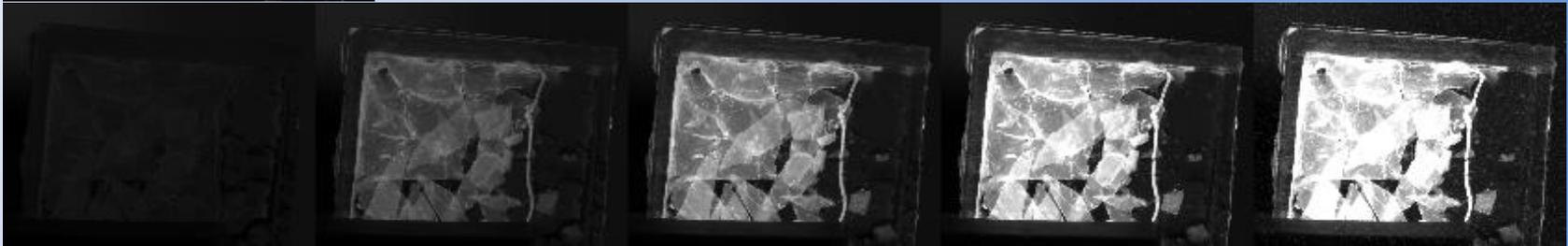
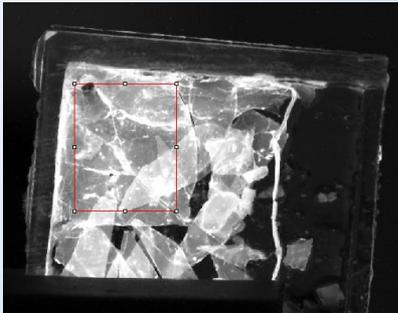


Photon Output Uniformity

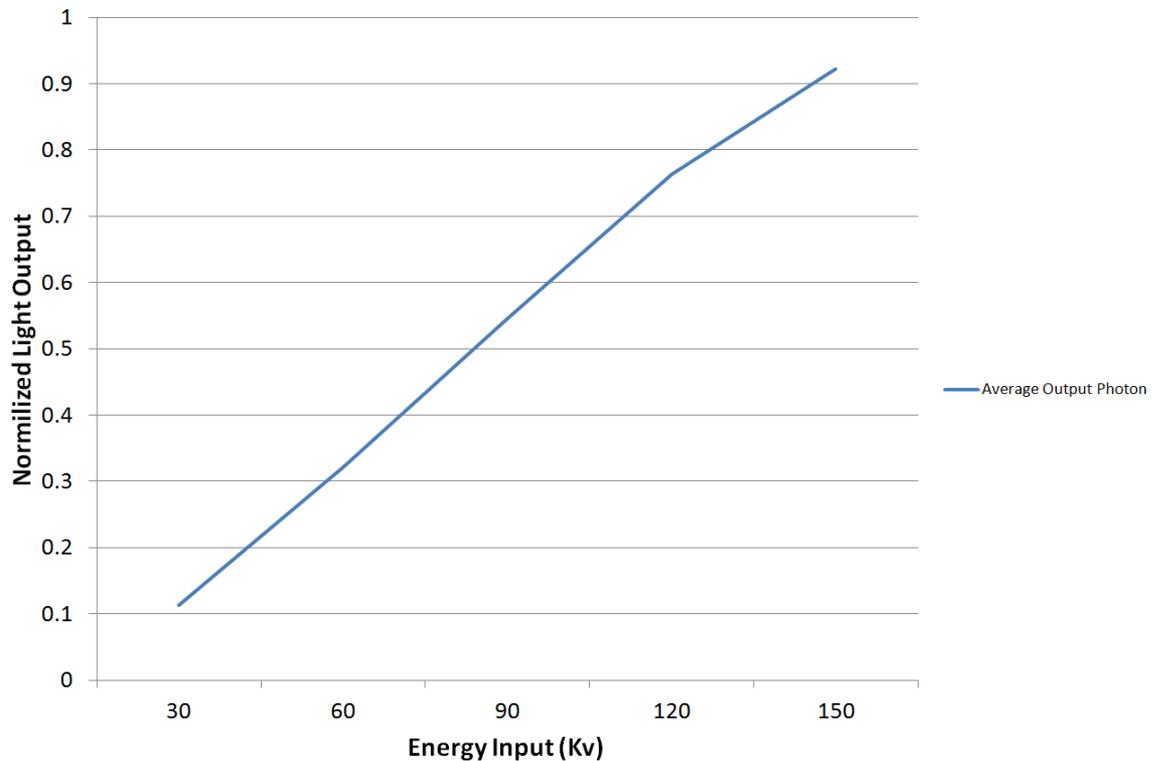
NASA Aeronautics Research Institute

- Standard Normalized Deviation of Photon Count:

- 30 Kv - 0.30%
- 60 Kv - 2.21%
- 90 Kv - 4.37%
- 120 Kv - 5.72%
- 150 Kv - 4.02%



Average Output vs Energy Input



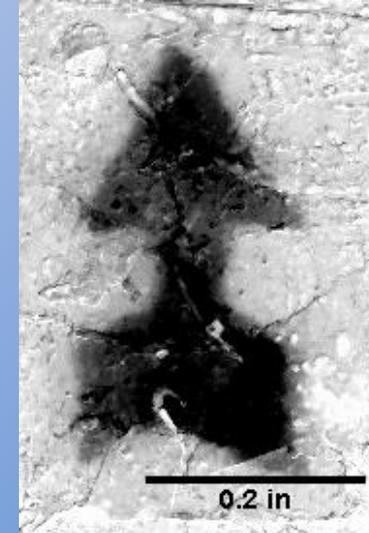
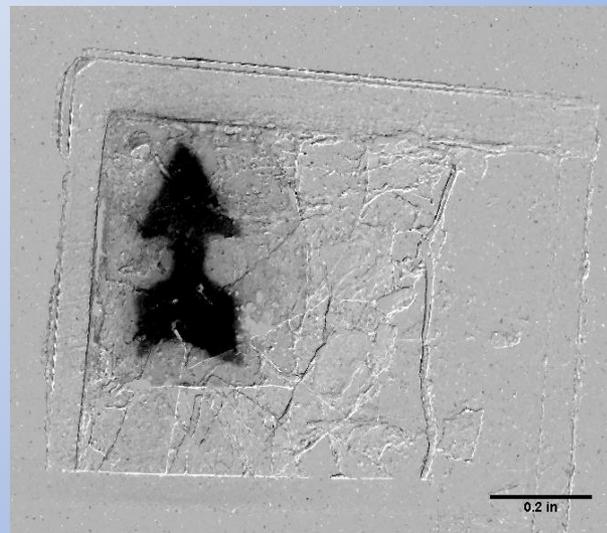
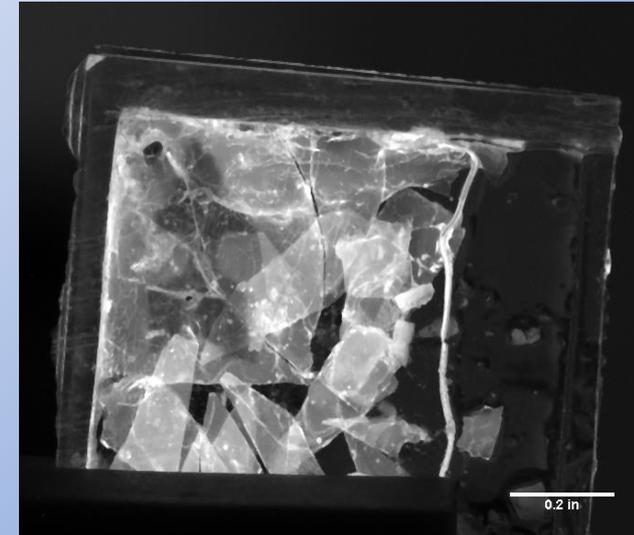
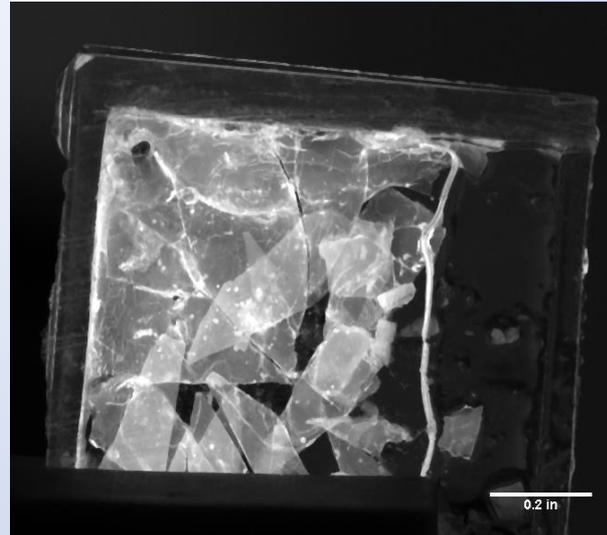


Results - Polymers

NASA Aeronautics Research Institute

• Several images were taken under x-ray excitation:

- Upper Left: Sample no target object.
- Upper Right: Subtracted Pb arrow target
- Lower Left: Sample with Pb Arrow target.
- Lower Right: Subtracted zoomed sample with Pb Arrow target.

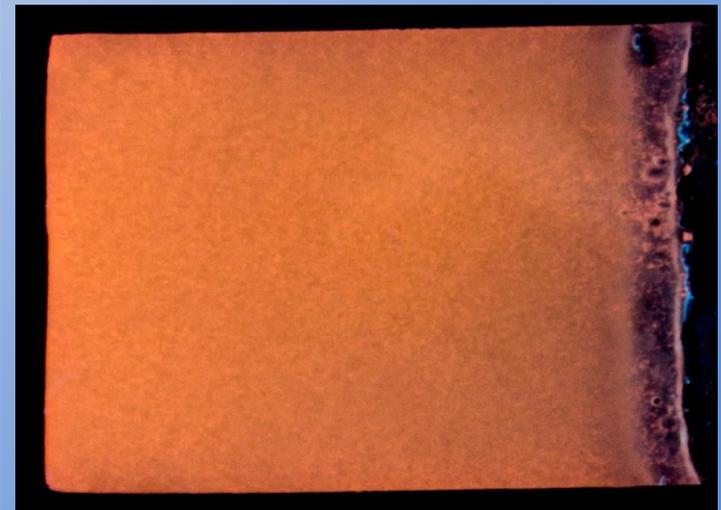
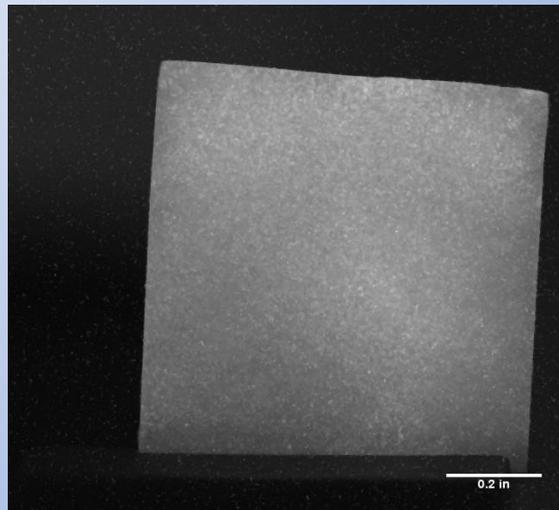
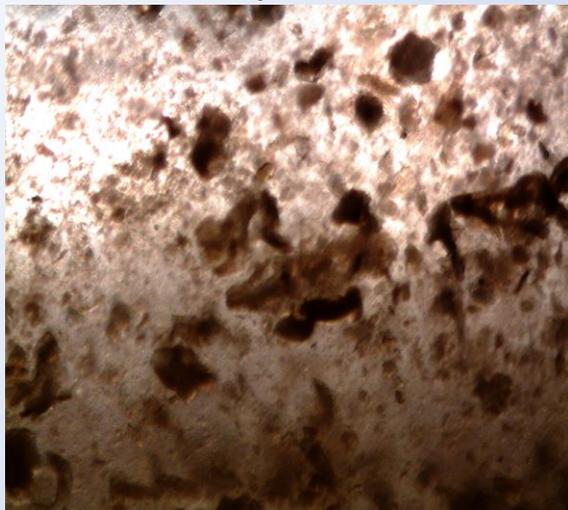
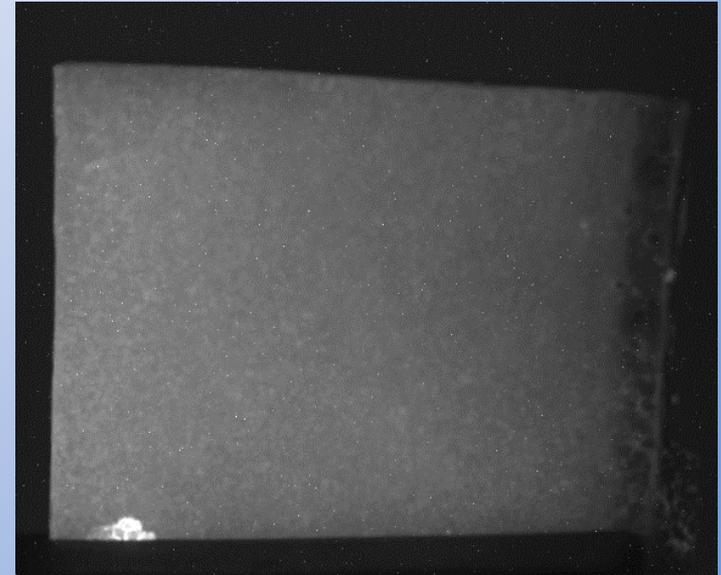




Results - Polymers

NASA Aeronautics Research Institute

- X-Ray characterization of the mixed polymer generates fairly uniform scintillation across the polymer.
- Lower left hand corner of the polymer was damaged but produced very highly directional light output.
- Cloudy nature of the sample is due to QDs prematurely crystallizing in the polymer forming small crystals (see below).

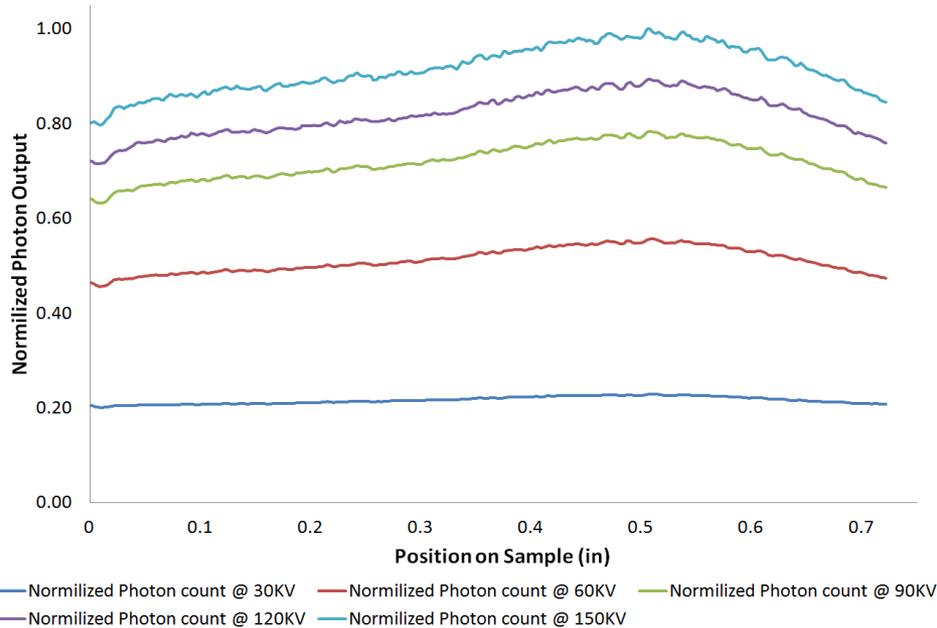




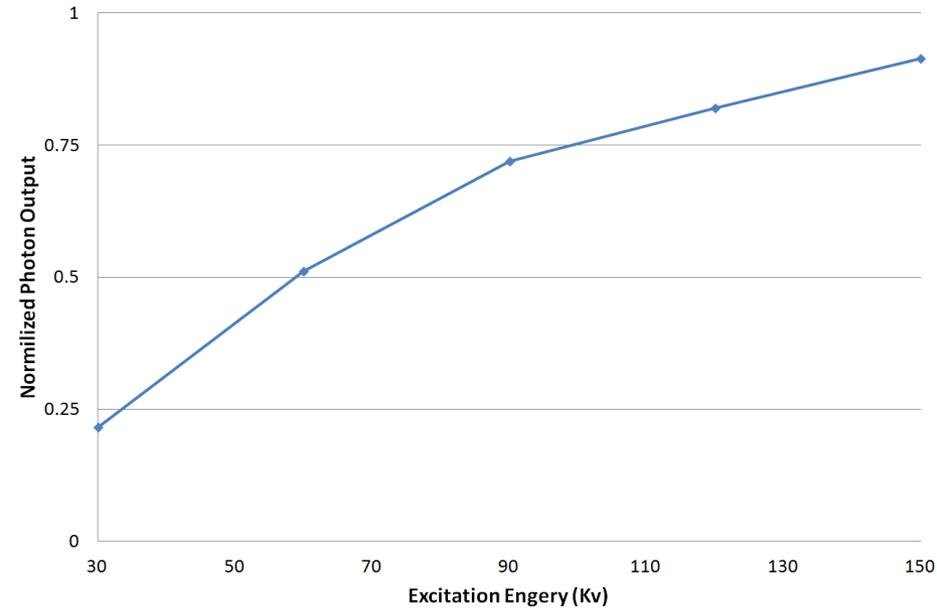
Photon Output Uniformity

NASA Aeronautics Research Institute

Normalized Photon Output vs Sample Position

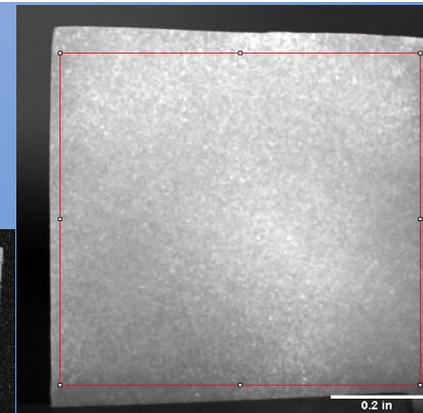


Average Output Photon vs Energy Input



- Standard Normalized Deviation of Photon Count:

- 30 Kv - 0.7%
- 60 Kv - 2.6%
- 90 Kv - 3.8%
- 120 Kv - 4.4%
- 150 Kv - 4.9%

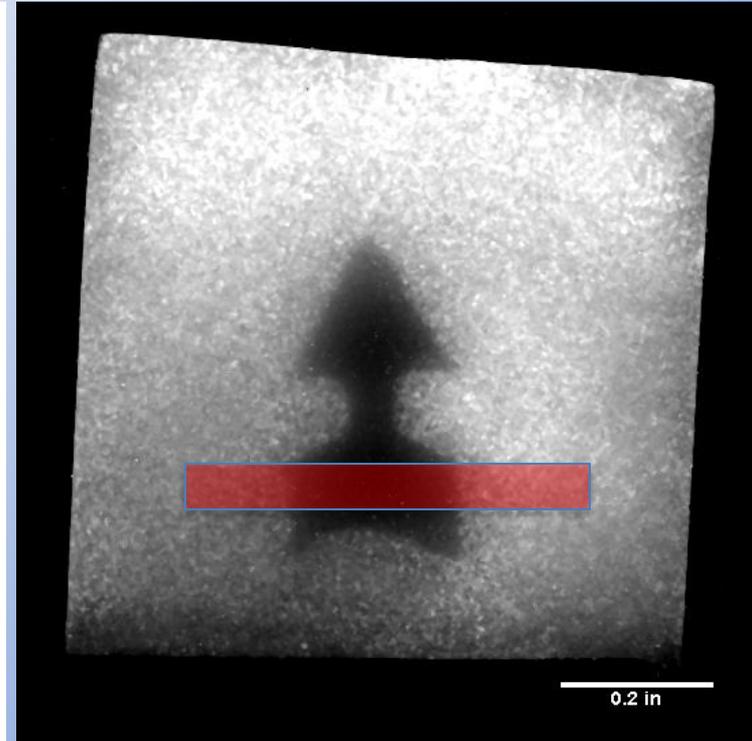
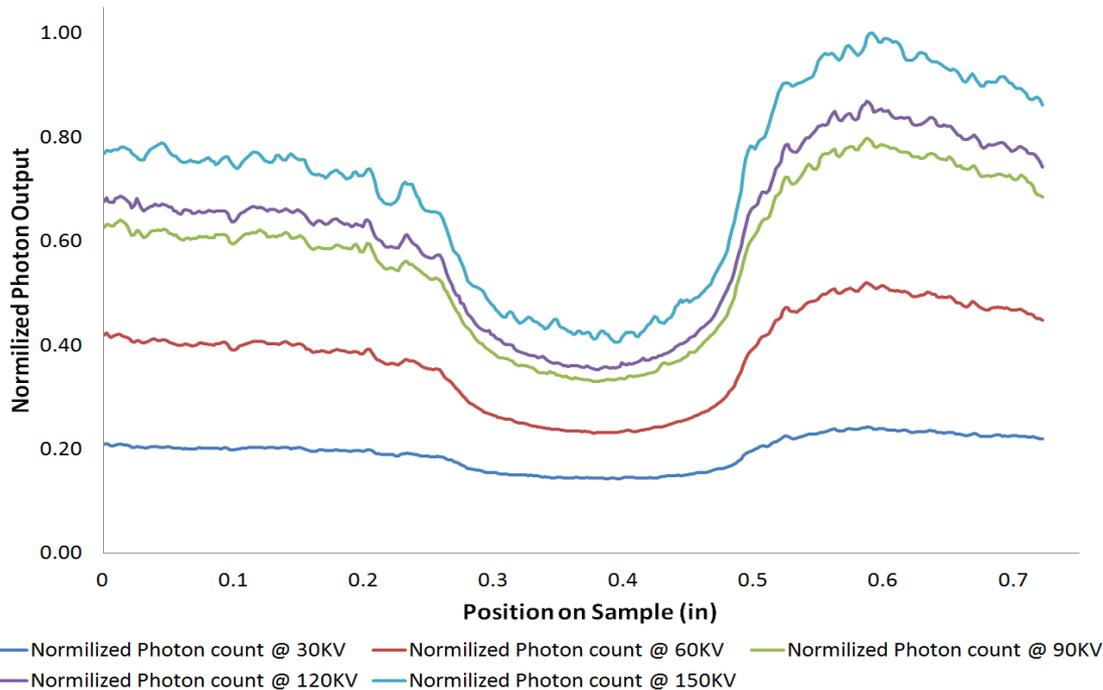




X-Ray Image Response

NASA Aeronautics Research Institute

Normalized Photon Output vs Sample Position



- Normalized Response Change:
 - 30 Kv – 9.93%
 - 60 Kv – 29.02%
 - 90 Kv – 46.91%
 - 120 Kv – 51.71%
 - 150 Kv – 59.47%

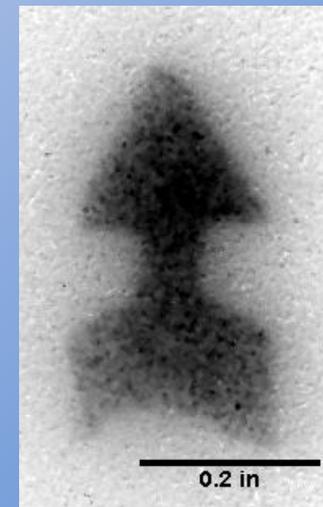
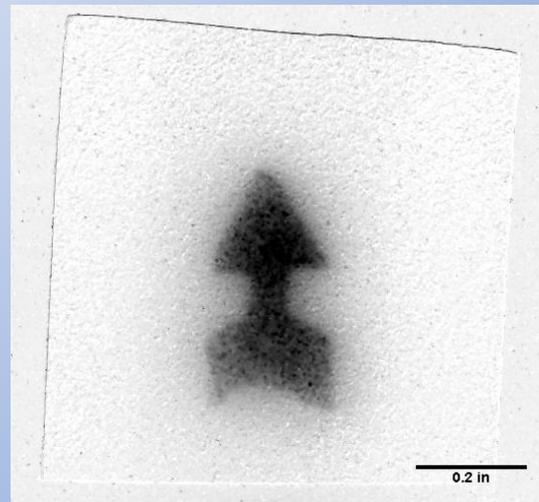
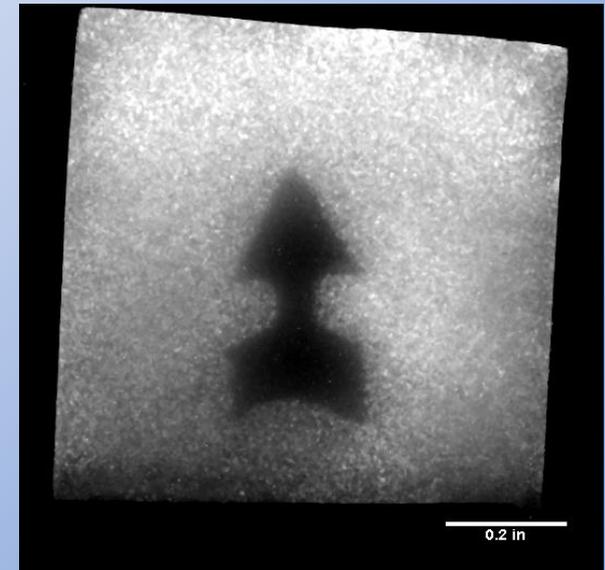
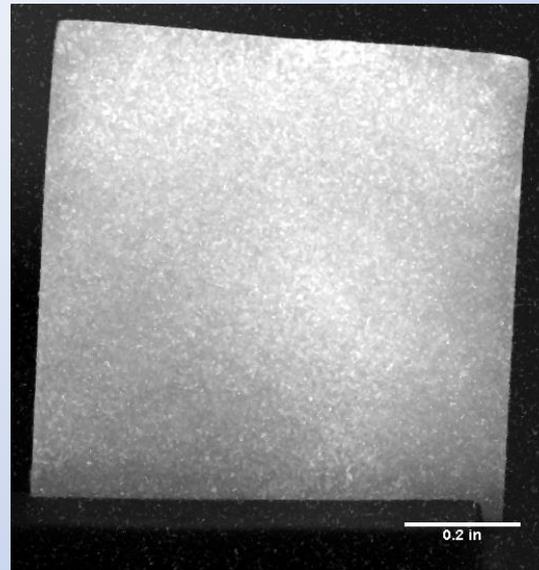


Results - Polymers

NASA Aeronautics Research Institute

Several images were taken under x-ray excitation:

- Upper Left: Sample no target object.
- Upper Right: Subtracted Pb arrow target
- Lower Left: Sample with Pb Arrow target.
- Lower Right: Subtracted zoomed sample with Pb Arrow target.

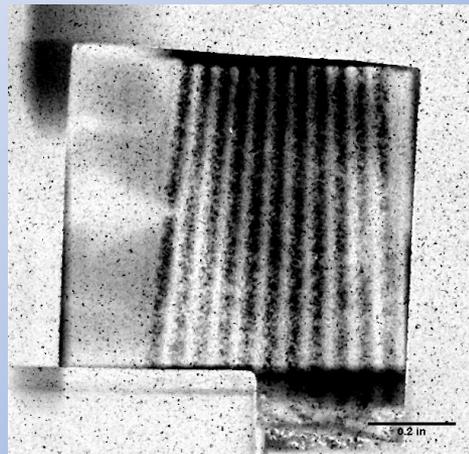
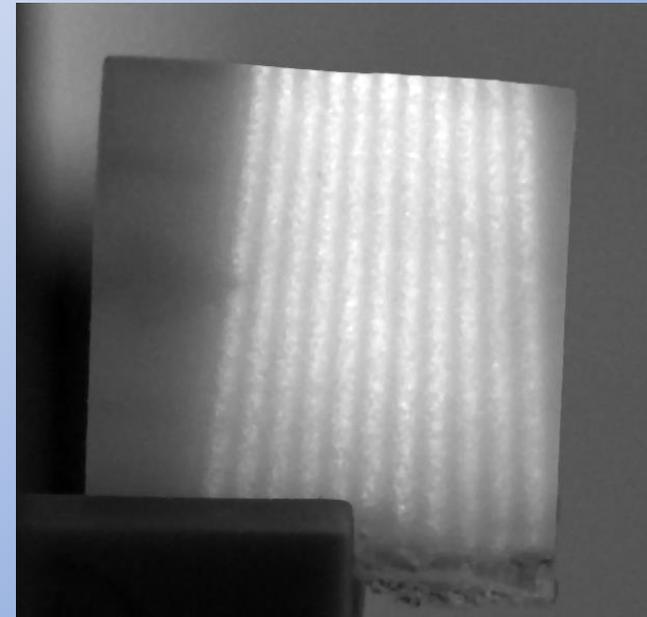
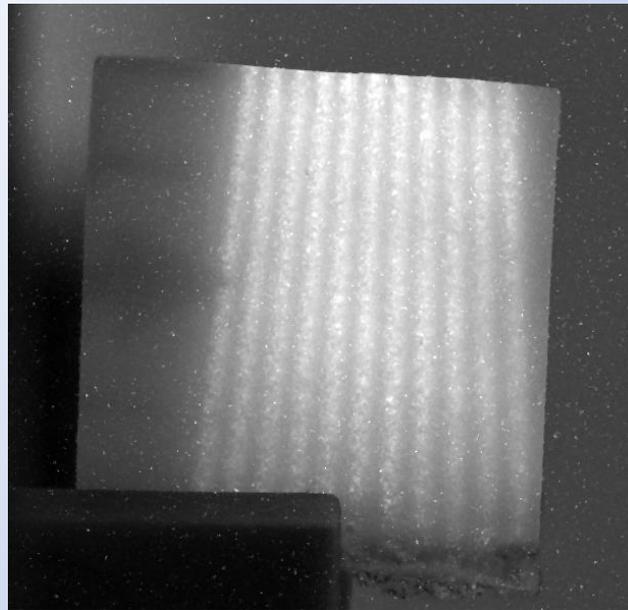




X-Ray Resolution Test Patterns

NASA Aeronautics Research Institute

- Images:
 - Upper left 120 Kv test pattern
 - Upper right 90 Kv test pattern
 - Lower left 120 Kv target arrow
 - Lower right test pattern
- Using a standard x-ray resolution test pattern shows resolution of the current system
- Target arrow can also be seen in both the 120 and 90 Kv results.





Phase 2

NASA Aeronautics Research Institute

- Perform fluorescence characterization of StQDs based nanocomposites (NC) in terms of quantum efficiency, spectral emission, fluorescent emission decay times and their dependence on temperature.
- Improve fabrication of StQDs based NC with better solution based processing techniques.
- Model and test X-ray absorption properties for StQDs based NC. Determine the energy range of X-ray detection sensitivity with StQDs based NC.
- Fabricate 2-D pixilated StQD samples for characterization of spatial resolution limit.
- Investigate the long term X-ray effect on stability and sensitivity
- Explore optical fiber geometry for x-ray detection.
- Explore use of StQDs for other radiation particle detection such as protons and electrons.
- Explore use of StQDs based NC for radiation shielding and detection.



Phase 2 - Budget

NASA Aeronautics Research Institute

- NASA LaRC NESB will be seeking continuation of phase 2 for 18 Month effort.
- Semi-formal quote has been established with NIA
- Mesolight has given confirmation to continue to support this project.

Proposal Title: Scintillating Quantum Dots for Imaging X-rays (SQDIX) for Aircraft Inspection		From:	05/01/14	
		To:	10/31/15	
PI's Name: Eric Burke				
NASA Center: LaRC				
<i>Use May 1, 2014 to Oct 31, 2015 for Period of Performance.</i>				
<i>Assume \$150K for 1 Full Time Equivalent of 2080 hours per NASA Civil Servant.</i>				
Budget Request (\$K)	<u>FY14</u>	<u>FY15</u>	<u>FY16</u>	<u>TOTAL</u>
Civil Servant Salary: Eric Burke LaRC	25	25	15	65
Total Civil Servant Travel	2	2	2	6
In-house Procurement	5	5	5	15
External Procurement - NIA	158.4			158.4
External Procurement - Mesolight	25	50	25.6	100.6
External Procurement - Hardware Consumables	8	15	7	30
Budget Total	223.4	82	54.6	375



Conclusions

NASA Aeronautics Research Institute

- Use of QDs for x-ray image production has not been well explored and is opening the door to lower cost high resolution x-ray devices.
- Development of SQDIX technology will directly effect the x-ray inspection methods for aircraft in the area of both metals and composites.
 - SQDIX could enable x-ray inspection of in service parts by enabling very high speed x-ray radiography.
 - SQDIX can enhance the x-ray resolution capabilities beyond the current technology.
 - SQDIX could greatly enhance the resolution and reduce the time to acquire computed tomography scans.
 - SQDIX will enable detector production methods that can scale to full vehicle radiography.
 - SQDIX enables the use of COTS light and camera optics allowing for variable resolutions and focusing without reconfiguration.
- Development of SQDIX could have major impacts on the medical community.
- Develop self-illuminating radiation shielding.